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Large Area Direct Fabrication of periodic Arrays using Interference Patterning

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Abstract

In this study, we introduce a recently developed approach for the fabrication of two and three dimensional structures using Direct Laser Interference Patterning (DLIP). Different periodic arrays were fabricated on polymers, resists and carbon like layers using a nanosecond pulsed laser system. The fabricated structures range from the submicrometer (~180 nm) to micrometer (~10 µm) range, and its resolution (minimal achievable feature size) depends basically on interaction nature of the laser light with the processed material, as well as the utilized wavelength and laser processing parameters. Different examples of fabricated arrays and their applications are discussed.

Keywords: Direct Laser Interference Patterning; Micro and submicrometer fabrication; Metals; Polymers; Ceramics

1. Motivation / State of the Art

Periodic patterned surfaces do not merely provide unique properties, but act as intelligent surfaces capable of selectively influencing multiple functionalities with applications in biomaterials [1-4], surface engineering [5, 6], photonics [7, 8] and sensor systems [9]. Numerous techniques have been explored and applied to fabricate such micro- and nano- features (e.g. nanoimprint lithography, laser writing, optical lithography, mask projection Excimer Laser lithography). However, only few of them are suitable for the fabrication of periodic structures on different materials in a single process step [10].

One of the more recent advances allowing fabrication of periodic arrays within the micro- and submicrometer scale involves Direct Laser Interference Patterning (DLIP) [11, 12]. This method is particularly suited to fabricate periodic patterns on planar as well as non-planar surfaces. Another significant advantage of this technique compared to other surface patterning methods is that fairly large areas can be processed within a short period of time (up to several cm^2/s) using a single or multiple laser pulses [13]. Hence, it offers a route to large-scale production.

By properly selecting the process parameters, this technique is well suited to create different surface topographies. Likewise, different effects such as melting, healing as well as defect and phase formation can be induced [12]. Therefore, electrical, chemical and/or mechanical surface properties can be periodically varied.

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2. Experimental

2.1. Materials

Photopolymerization experiments: negative SU-8 and positive AZ-1505 photoresists (manufactured by MicroChem and Microchemicals, respectively) were utilized. Prior to spin coating the surface of the wafers was thoroughly cleaned with ethanol and heated for 2 min to dehydrate the substrate and improve the adhesion. The resist was then spin coated on silicon wafers with a thickness of 0.5 and 1.5 µm. The thickness of the resist layer was controlled by adjusting the spinning speed (between 2000 and 6000 rpm). Before exposure, the samples were pre-baked on a hot plate for 1 to 4 min at 95ºC, depending on film thickness. After exposure, they were post-baked on hot plate for 4 min at the same temperature. After a minute for relaxation time, the samples were developed using a specially designed developer, which basically consists of propylene glycol methyl ether acetate (PGMEA, Y020100 from MicroChem). Finally ethanol was used to rinse the structure and remove residual developer for approximately 15 s.

TaC film preparation: the tetrahedral amorphous carbon thin films were prepared on polished single crystal silicon substrates with a filtered cathodic arc equipped with a 90° filter tube with three magnetic coils. The magnetic filter reduced the number of particles that reaches the substrate. Ta-C films with a thickness of a few micrometer were produced by an unfiltered arc.

Polymer substrate: Polycarbonate (PC), and polyimide (PI, Kapton®) films were purchased from Goodfellow GmbH (Bad Nauheim, Germany) and used as received. The films were thoroughly washed with detergent in water, and then rinsed with ethanol, to remove any surface contaminants present.

2.2. Laser Interference

The laser interference experiments were carried out using a Nd:YAG laser with 10 ns laser pulses at 266 or 355 nm wavelength (λ). The primary laser beam was split into two beams, which were then recombined to interfere on the substrate's surface (Figure 1). In this way, a periodic intensity distribution is obtained [12]. The grating period (Λ) can be adjusted by varying the angle of incidence between both laser beams (α). Both an adjustable aperture and a telescope system were used to control the desired beam size. A mechanical shutter was used to ensure that only a predefined number of laser pulses passed to hit the substrates.

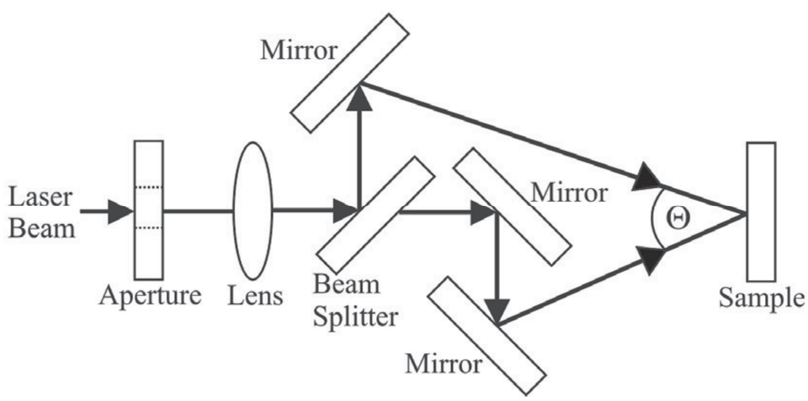

Figure 1. Schematic experimental setup for the interference experiments showing the optical components required to split and guide the laser beams to the sample.

2.3. Tribological characterization

The tribological performance of the films was measured with a reciprocating ball on disc method (CSM Tribometer) under ambient conditions. 100Cr6 balls with a diameter of 6 mm, coated with 1 µm ta-C were utilized. The maximum linear speed was 3 cm/s, the normal load 1 N and the sliding distance 1000 m, corresponding to 1/2 amplitude per cycle of 0.5 mm and 10^6 cycles. Prior to experiment the test specimens were cleaned with acetone in an ultrasonic bath and dried at ambient temperature. After cleaning the substrates the lubricant 10W60-oil was deposited on the substrates.

2.4. Surface characterization

A scanning electron microscope (SEM, operating voltage: 5 kV) and an atomic force microscope (AFM) were used to analyze the surface topography of the irradiated substrates.

3. Results and Discussion

3.1. Laser Interference Lithography (periodic local photo-polymerization)

Laser interference patterning was utilized to locally interact with photoresist films (SU-8 and AZ-1505) on Silicon substrates. After irradiation of the sample (355 nm), the more exposed and less exposed parts present different solubility in the developer, leading to a porous photoresist structure. In the case of a negative photoresist (e.g. SU-8), the regions corresponding to the interference maxima are photo cross-linked and thus remaining attached to the substrate after development. In this way, stiction problems can be avoided [14]. Using a two-beam setup (Figure 1), line-like arrays with a spatial period of 1500 nm were fabricated as shown in Figure 2a. However, for the fabrication of 2D or 3D structures at least three single coherent laser beams are required. Besides this method, a more robust technique consists of performing a double exposure process by rotating the substrate a specific angle between two consecutive irradiation steps [11, 13]. In this way, mechanical stable cross-like structures can be fabricated as shown in Figure 2b. A layer by layer approach can be also utilized for the fabrication of bio-inspired structures. For the example depicted in Figure 2c, a thin layer of the photoresist was irradiated twice with a lattice period of 500 nm and rotation angle of 60° to generate the lower sub-micrometer hexagonal pattern. After that, a second layer of the photoresist was spin-coated over a previously patterned and developed layer, and then irradiated with line-like interference patterns but with a lattice period of 5.0 µm. The structure in this way fabricated is very similar to a natural inorganic structure of a diatom (C. walesii) [15, 16], which shows photonic properties.

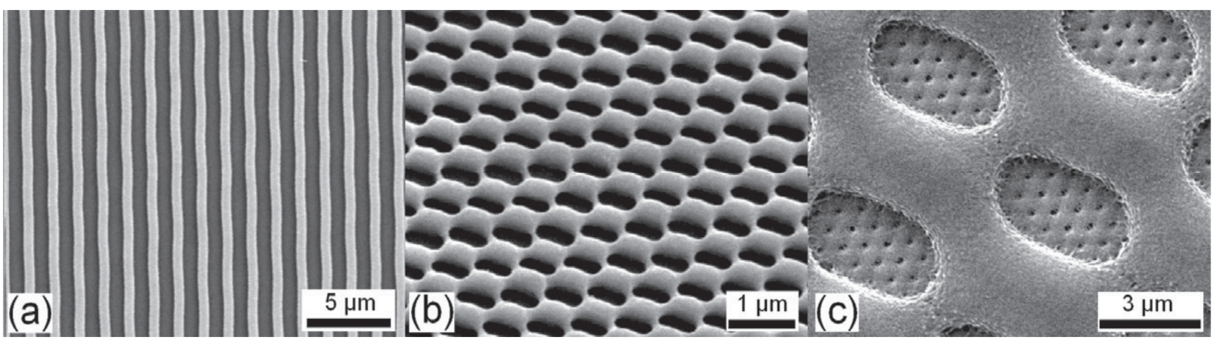

Figure 2. Fabricated (a) line (Λ = 1500 nm), (b) lattice (Λ = 500 nm) and (c) hierarchical structure (Λ = 5µm and 500 nm) on SU-8 resist [16].

In contrast to negative resists, in a positive photoresist (here AZ-1505) the exposed parts from the polymer dissolve into the developer after irradiation of the sample [17]. In consequence, the double exposure strategy described above leads to the fabrication of isolated dot structures with different arrangements if sufficient energy is applied (Figure X.b). On the other hand, at lower exposure doses the resist is partially dissolved into the developer after irradiation at the interference maxima positions (Figure 3b). However, at the intercepting positions corresponding to the overlap of two interference maxima from the two consecutive irradiation steps (but with rotation of the sample in between, in this case 90°), the exposure dose is doubled and thus the material is totally removed (Figure 3b). Thus, by properly controlling the exposure dose, also 2.5D structures can be fabricated (Figure 3b).

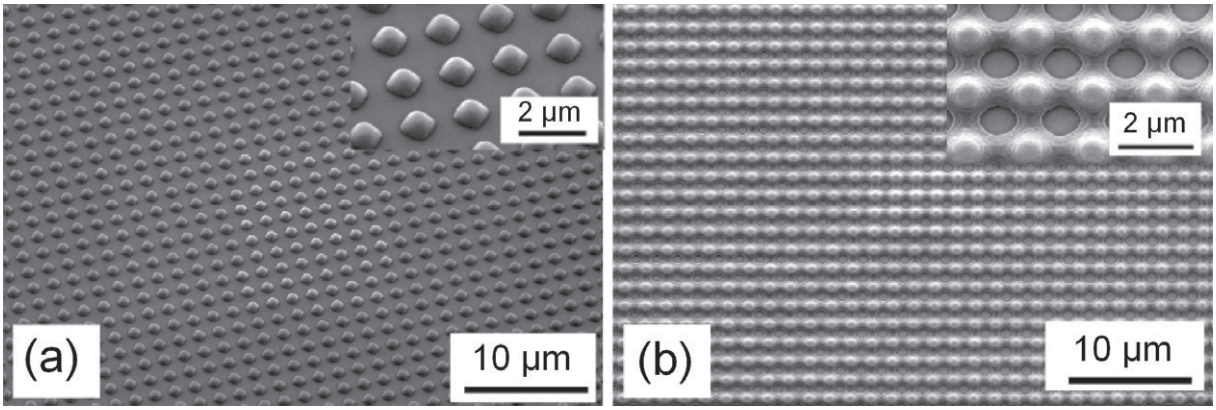

Figure 3. Two dimensional structures on AZ-1505 with Λ = 1.7 µm (film thickness 500 nm). (a) Isolated structures fabricated with F = 21 mJ/cm² and (b) 12 mJ/cm². In both cases the samples were rotated 90° between exposures of the resist.

3.2. Patterning of polymer substrates

Using the setup described in section 2.2, we fabricated periodic arrays on commercial polystyrene (PS) and polyimide (PI) substrates. Due to the low absorption of PS-substrates at both 355 and 532 nm wavelengths, the quadruple harmonic wavelength (266 nm) of the laser system was used. As it can be observed in Figure 4a-b, well defined periodic arrays could be fabricated. The ablated regions at the material's surface correspond to the interference maxima positions, where the threshold fluence is surpassed. Despite the short wavelength (266 nm) used to irradiate the PS substrates, a significant contribution of photothermal excitation is expected [18]. This means that the material is first melted before being ablated. In consequence, the obtained surface profile is quite smooth as shown in Figure 4b. Apart from that, the structure depth for single pulse irradiation can be varied with the laser fluence. The maximal achievable structure depth increases with increasing the spatial period up to values which are close the to absorption depth at the utilized laser wavelength.

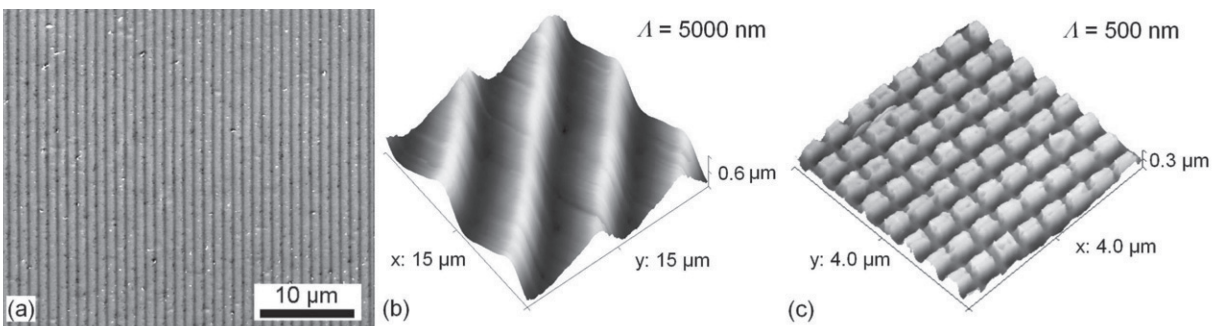

Figure 4. Fabricated line-pattern on (a) PS with Λ = 1.0 (SEM picture) and (b) 5.0 µm (AFM). (c) Lattice-structure on PI with Λ = 500 nm (AFM).

To fabricate grid-like structures, the polymer substrate can be rotated a specific angle between two consecutive irradiation steps. This process is shown for PI substrates, in Figure 4c, for a rotation angle of 90°. The laser fluence utilized in this case was 0.5 J/cm². PI polymer presents a high absorption at both 266 and 355 nm wavelengths [19]. Moreover, compared to PS, sharper edges were observed (even for 500 nm arrays, see Figure 4c). Preliminary results of bacterial adhesion test on these patterned polymers have demonstrated that by controlling the topographical characteristic (period and pattern shape) it is possible to fabricate antibacterial surfaces based only on topographical effects, without changing the surface chemistry [20].

3.3. Patterning of diamond like carbon films

Diamond like carbon films present attractive properties such as high hardness, low friction and wear, as well as excellent biocompatibility. Due to high absorption of the films at different wavelengths, these materials films can be easily patterned. Depending on laser processing parameters and layer thickness, different effects can be observed. At low laser intensities, the layers are graphitized which means an increase of the sp2 content [21]. Higher laser intensities produce delamination of the layer together with the formation of graphitic clusters in addition to a crystalline structure. However, by using a periodic variation of laser intensity, like in an interference pattern, these effects can be induced locally at the positions corresponding to the interference maxima positions.

For example, in Figure 5, graphitization, delamination and ablation of ta-C thin films are shown (60% sp3 content). The spatial period (Λ) was in this case 5.0 µm. Another example is depicted in Figure 6a, where 180 nm arrays were fabricated with 266 nm of laser wavelength. Also in this case, the material corresponds to ta-C films with 60% of sp3 content. In the last case, the film was locally graphitized at the intensity maxima. As application example, the tribological properties of ta-C can be improved. In comparison with untreated steel surfaces, the friction coefficient can be reduced up to 83% (absolute) as shown in Figure 6b.

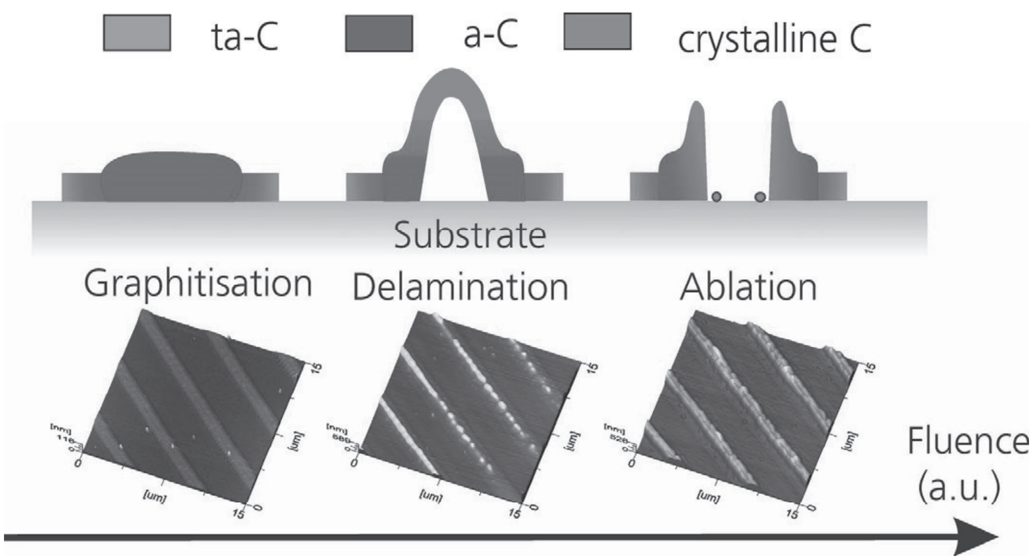

Figure 5. Schematical representation and experimental results of DLIP induced graphitization, delamination and ablation of ta-C. The DLIP experiments (period 5 µm) were performed at 48 nm thick ta-C with laser fluences of 38, 75 and 90 mJ/cm², respectively.

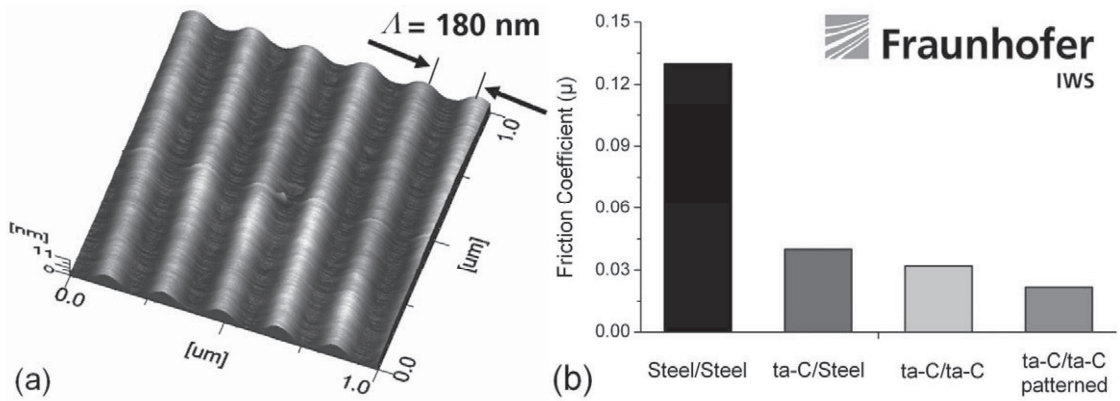

Figure 6. (a) Patterned ta-C layer with Λ = 180 nm. (b) Fric. coeff. of steel/steel, steel/ ta-C, ta-C/ta-C, ta-C/ta-C patterned (lubricated conditions).

4. Summary

Direct Laser Interference Patterning has been demonstrated to be a potential method for the direct fabrication of periodic arrays on different materials. Based on the material's properties as well as the laser parameters, different effects can be induced over the material's surfaces. The obtained structures can enhance different applications, for example friction and wear reduction on patterned ta-C films.

Acknowledgments

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